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Research Article

Pyrolysis of Microalgae *Chlorella sp.* using Activated Carbon as Catalyst for Biofuel Production

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Abstract

Microalgae, as a potential raw material for biofuel, has several advantages compared to other biomass. One effective way to convert microalgae into biofuel is by thermal cracking or pyrolysis, and using a catalyst or not. So far, studies on the use of microalgae, that are converted into biofuels, is still use highly concentrated catalysts in packed bed reactors, which is not economical. Therefore, the aim of this study is to convert *Chlorella sp.* into biofuels with conventional pyrolysis without and using an activated carbon catalyst using packed bed reactor with bubble column. The reaction temperature is 400–600 °C, pyrolysis time is 1–4 hours, and the active carbon catalyst concentration is 0–2%. The 200 grams of *Chlorella sp.* and the catalyst was mixed in a fixed bed reactor under vacuum (–3 mm H₂O) condition. Next, we set the reaction temperature. When the temperature was reached, the pyrolysis was begun. After certain time was reached, the pyrolysis produced a liquid oil product. Oil products are measured for density and viscosity. The results showed that the conventional pyrolysis succeeded in converting microalgae *Chlorella sp.* into liquid biofuels. The highest yield of total liquid oil is obtained 50.2 % (heavy fraction yield, 43.75% and light fraction yield, 6.44%) at the highest conditions which was obtained with 1% activated carbon at a temperature and pyrolysis time of 3 hours. Physical properties of liquid biofuel are density of 0.88 kg/m³ and viscosity of 5.79 cSt. This physical properties are within the range of the national biodiesel standard SNI 7182-2012. The packed bed reactor completed with bubble column is the best choice for converting biofuel from microalgae, because it gives different fractions, so that it is easier to process further to the commercial biofuel stage.

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Keywords: Pyrolysis; Microalgae; *Chlorella sp.*; Activated carbon; Biofuel

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1. Introduction

Increasing energy demand is a serious problem in various countries in the world, because it has an impact on all sectors, such as: social development, development and the country's economy [1]. Furthermore, the International Energy

Agency (IEA), states that the world population is expected to increase from 7.4 billion in 2016 to 9.1 billion in 2040 therefore, global energy must grow at least 30% in the next 25 years [2]. Fulfillment of energy is still focused on petroleum fuels whose utilization contributes significantly in producing greenhouse gas emissions which can be global warming and climate change [3]. Therefore, it is necessary to find alternative energy that is environmentally friendly and sustainable energy sources at lower price

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es. Some alternative energy sources come from geothermal, solar, wind, and water power [4]. At present there are around 14% of the total world energy supplied by renewable energy [5]. However, some of these alternatives have not been able to replace in solving the global energy crisis because of its form [6]. New alternative energy sources must be in liquid form to replace petroleum fuels so that they are able to replace petroleum fuels and biomass is believed to be able to become cheap, sustainable and environmentally friendly biofuels [6].

Biomass has also been developed from the first generation to the third generation [7]. First generation biomass, which originates from food crops, has caused several controversial issues such as food prices, deforestation, global warming and threats to biodiversity [8]. Whereas the second generation, related to the technology, fuels that use lignocellulosic biomass are still not operating and the future of their development is uncertain. Meanwhile, most technology is needed to produce bioenergy from agricultural crops, trees or organic waste; must be able to operate, although efficiency can still be improved and its supply need a large area [9]. Because of this, algae are presented as third generation biomass. Algae are aquatic organisms and are categorized as macroalgae (e.g. seaweed), cyanobacteria (unicellular bacteria) and microalgae (prokaryotic or eukaryotic photosynthetic microorganisms) [10].

Microalgae as an alternative biomass from biofuels has many advantages over the previous generation, including: (1) very high biomass production [11], 5-30 times that of other oil-producing plants in the same region; (2) there is no competition from the food sector; (3) can be developed (cultivated) in non-arable land or wastewater; and (4) lipids (oil) produced can reach 60% of their dry weight in some species. *Chlorella sp.* (this work) one of the microalgae with high bio-oil yields, up to 28.60% [3].

Therefore, in recent years, many methods have been carried out to convert microalgae into biofuels. This method can be broadly divided into three categories, namely chemical reactions (extraction-trans-esterification, trans-esterification of situ), bio-chemical techniques (fermentation, Algal Lipid Upgrading). and thermo-chemical techniques (combustion, gasification, and pyrolysis) [12-15]. However, pyrolysis techniques are often used for more than 50 years and provide high quality results and lower prices compared to other techniques [10]. Hu *et al.* changed *Chlorella vulgaris* with mi-

crowave assisted pyrolysis processes on various catalysts, activated carbon, CaO, SiC, and solid residues from products. The highest yield of biofuel was 87.47% in the use of 5% active carbon catalysts, while the use of solid residue catalysts from products obtain the second best results after activated carbon [14]. In addition, Chaiwong *et al.* [16] have converted the microalgae *Spirulina sp.* into biofuels by pyrolysis techniques at various temperatures (450-600 °C) over a fixed bed reactor under atmospheric nitrogen without catalysts. The results showed that the optimum temperature of bio-char and biofuel is achieved at 500 and 550 °C, respectively. Therefore, the aim of this study is to convert *Chlorella sp.* into biofuels with conventional pyrolysis without and using an activated carbon catalyst over a packed bed reactor with bubble column to separate the liquid product.

2. Materials and Methods

2.1 Materials

Microalgae *Chlorella sp.* (dry weight) is obtained from Badan Perikanan Budidaya Air Payau (BBAP) Situbondo, East Java (Table 1) and activated carbon catalyst (0%, 1%, 2%) (Table 2).

2.2 Experimental Apparatus

The main equipment used in this study is the same with used by Thahir *et al.* [17] that consist of a fixed bed reactor made of stainless steel with a maximum heating of 750 °C, which completed with an insulated chamber to reduce heat emission from the exit system (Figure 1). The temperature was measured using and controlled by an electric source producing heat using an integral (PI) with an off-set of 2 °C. The heating speed was 15.46 K/min, so that it is categorized as slow pyrolysis under vacuum pressure ($P_{\text{input}} = -7 \text{ mmHg}$; $P_{\text{out}} = -10 \text{ mmHg}$) to isolate from the presence of oxygen.

2.3 Experimental Procedures

In this study, the reactor temperature used was 450-600 °C, pyrolysis time was 1, 2, 3, and 4 hours, and activated carbon concentration was 0%, 1%, and 2%. Firstly, 200 g of *Chlorella sp.* was put into the fixed bed reactor. Secondly, thermometer and vacuum pump was installed on the device. Furthermore, the temperature was adjusted according to the variables set in the initial design of this study. The final step, the products formed in the form

of liquid and gas phases during the process were accommodate. The liquid phase product, as the result of condensation of pyrolysis vapors that passes through the condenser consisting of two layers, were mutually separated namely heavy fraction and light fraction. The condensation process of the gas formed in two stages. First, it was in the liquid condensate reservoir 1 (tray 1), and the second stage was in liquid condensate reservoir (Tray 2). The not condensed gas products were flowed to the gas

washing bottle accommodated by the gas reservoir. The liquid product is evaporated to remove the water content formed during the process. The liquid product formed on tray 1 is called light fraction liquid, while that in tray 2 is called heavy fraction liquid. The final step is to analyze the yield of each product.

2.4 Yield Calculation

The mass percentage of the composition of the product formed can be calculated to obtain % conversion (liquid, gas, and char) according to the following equations:

a. Liquid product (bio-oil):

$$\% \text{yield bio-oil} = \frac{\text{liquid mass}}{\text{microalgae mass}} \times 100\% \quad (1)$$

b. Char product:

$$\% \text{yield char} = \frac{\text{char mass (solid residue)}}{\text{microalgae mass}} \times 100\% \quad (2)$$

c. Gas product:

$$\% \text{yield gas} = 100\% - (\% \text{yield bio-oil} + \% \text{yield char}) \quad (3)$$

2.5 Fourier Transformed Infrared (FTIR) analysis

Infrared spectrum was used to qualitatively determine the functional groups of bio-oil (Thermo Scientific FTIR spectrum, Nicolet IS10, with a spectral range 7800–350 cm^{-1}). The bio-oil sample was dropped on the plate provided on the device which was then pressed using a mechanical device. This pressure was maintained for several minutes and then the plate was placed in the FT-IR sample holder for analysis.

3. Results and Discussion

3.1 Effect of Temperature on Pyrolysis Product

Yield (liquid, bio-char and gas) from conventional pyrolysis products is presented in Figure 2, which aims to determine the optimum temperature used in this study by varying the tem-

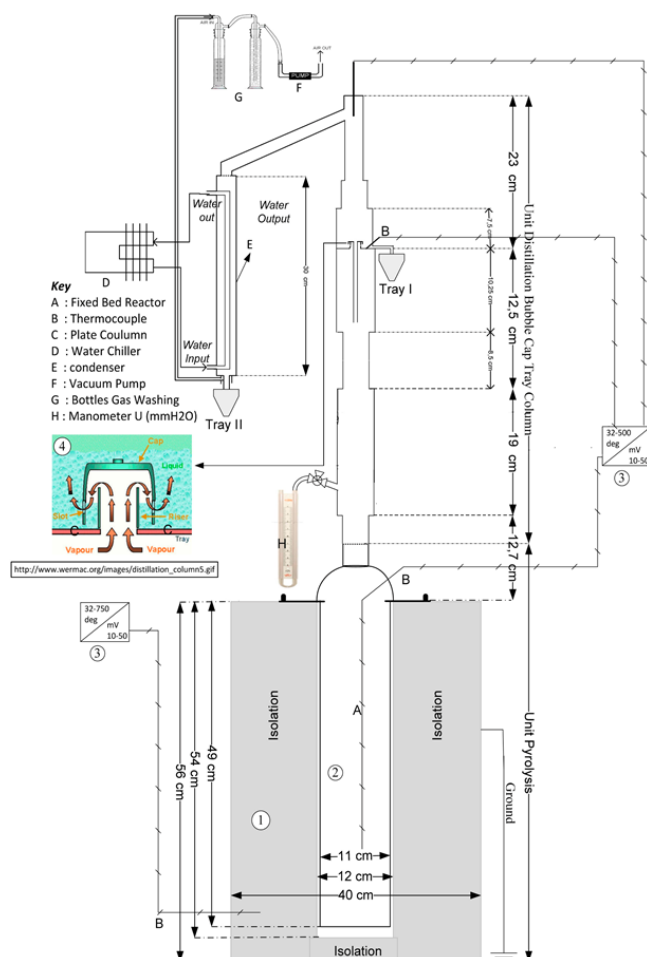


Figure 1. Schematic of conventional pyrolysis apparatus with fixed bed reactor.

Table 1. Proximate analysis of *Chlorella sp.*

| Component | Analysis result, % |
|-------------------|--------------------|
| Ash | 53.08 |
| Protein | 12.13 |
| Lipid | 12.41 |
| Rough fiber | 0.14 |
| Carbohydrate | 19.50 |
| Calorie (Kcal/Kg) | 1398.98 |
| Water content | 3.62% |

Table 2. The characterization of granule activated carbon.

| Characteristics | Result | Unit |
|---|---------------------|------------------------|
| Surface area | 1.95×10^6 | m^2/Kg |
| Pore volume | 10.28×10^2 | m^3/Kg |
| Diameter pore | 21.6 | \AA |
| Dielectric loss tangent ($\tan \delta$) | 1.71 | |
| Dielectric loss factor (ϵ'') | 27.50 | |
| Dielectric loss constant (ϵ) | 16.12 | |

perature of 450, 500, 550, and 600 °C for 4 hours and without using a catalyst.

Based on Figure 2, it shows that the higher the temperature, the percentage of total liquid yield (yield of light and heavy fractions) is increasing. This is because at high temperatures carbon chains would be more easily swelled than at low temperatures [18]. Furthermore, this is indicated because when the temperature becomes higher, the heat absorption capacity of microalgae becomes stronger, and then the whole temperature of the material becomes higher, so that the decomposition of macromolecules into micro-molecular products is easier to occur, therefore char is reduced and liquid yield increases. In addition, higher temperatures easily produce smaller and more profitable molecules to produce liquid products [19].

The highest percentage of total liquid yield was obtained at 550 °C at 42.81% and a decrease of 16.8%. This is because at higher temperatures, the gas produced is more than the liquid yield. The temperature has an important influence on product yield. At higher temperatures, the gas yield would be more numerous, the yield of liquid yield would increase to some extent then decrease, while the yield of solids (char) tends to be low. The higher the temperature, the heat value of the gas yield also increases, and the yield of solids tends to be constant, while the yield of liquid would decrease [19]. Based on the study of previous study, the high temperature also had an impact on the decrease in liquid quantity accompanied by high gas products. This is caused by the secondary cracking process which breaks long chains of organic compounds [20]. Based on this study, the highest char yield is at a temperature of

450 °C of 65.71% and at the next temperature rise the char yield is quite constant or there is not too significant difference. Therefore, the highest and smallest gas yields were 18.21% (at 600 °C) and 1.14% (at 550 °C), respectively. Based on these data, a temperature of 550 °C is used as a reference to get the highest liquid yield.

3.2 Effect of Temperature on Pyrolysis Product

Pyrolysis time is a parameter that has a significant role in the conversion of microalgae *Chlorella sp.* (dry weight) into biofuel products. Figure 3 shows that the longer the pyrolysis time, 1 to 3 hours, the higher the percentage of total liquid yield obtained. The longer of pyrolysis time would give the greater percentage of liquid yield. That is because the longer the time of pyrolysis, the contact between the reacting substances would be even greater so that the liquid yield obtained would be even greater. Another reason that supports the results of this study is the use of pyrolysis reactors which provide a large thermal effect so that the length of time would increase the temperature of the media so that the contact time would be greater as well and the % yield of liquid oil produced would increase as well. The results of this study are supported by previous study which states that the time variable influences the yield [21].

The best results are achieved on the use of 1% active carbon catalyst for 3 hours which is equal to 50.20% and tends to be constant thereafter (4 hours). This is due to the pyrolysis has reached equilibrium conditions so that the %total liquid yield would not increase, it may not even form a heavy fraction [22].

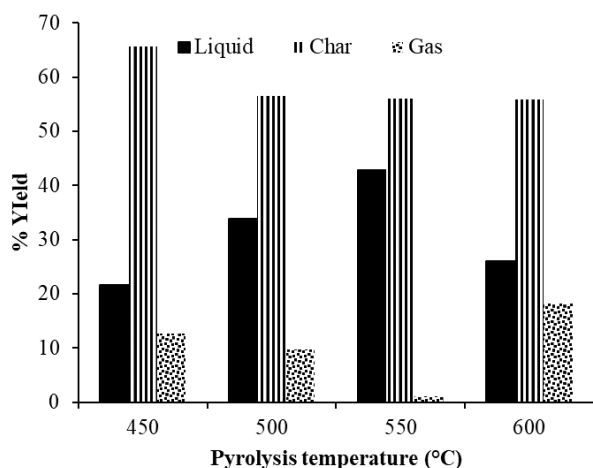


Figure 2. Effect of temperature on yield product of pyrolysis without catalyst.

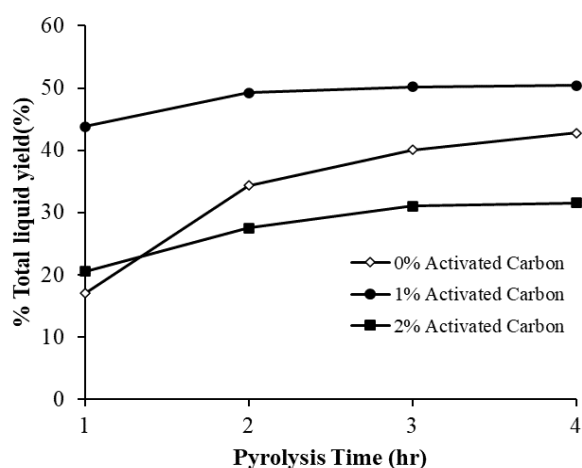


Figure 3. Pyrolysis time effect to %total liquid yield on various catalyst concentration.

Therefore, the pyrolysis time of 3 hours, pyrolysis has reached equilibrium conditions and is the optimum reaction time. So that it can be stated if with a longer reaction time, making biofuels becomes inefficient. This statement is supported by Patil *et al.* [23], which states that if the pyrolysis time is too long resulting in overheating in the reaction, so that a lot of gas is formed with a large amount, the formation of char, and the loss of energy in the process. The optimum time of the two fractions (heavy and light fractions) is 3 hours each yielding 43.75% (1% catalyst) and 10.37% (2% catalyst) and tends to be constant thereafter (Figure 4).

3.3 Effect of Catalyst on Pyrolysis Product

The effect of the catalyst in this study was reviewed at 550 °C and the optimum pyrolysis

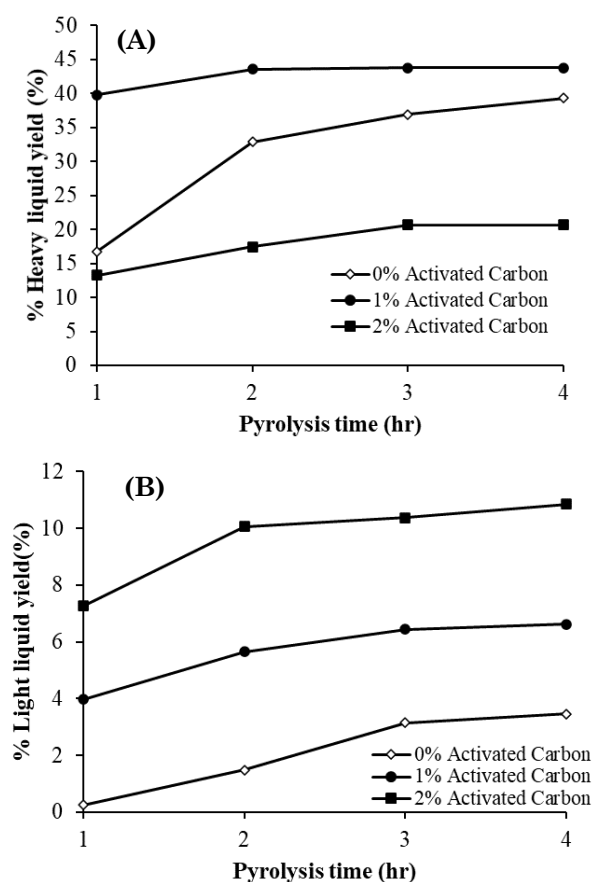


Figure 4. Heavy liquid (A) light liquid (B) yield function pyrolysis time at various catalyst concentration.

time was 3 hours. Based on Figure 5, it is known that the optimum catalyst is obtained when the use of activated carbon 1% with a yield of 50.20% with a percentage increase of 7.54%. This is because the catalytic effect on activated carbon catalysts occurs on the entire carbon surface. Therefore, the glycerol decomposition during the degradation process can produce a large vapor (product) due to decrease in activation energy by an activated carbon catalyst. The greater the amount of catalyst used, the greater the contact area of the catalyst used in the microalgae degradation process. It can decrease the activation energy, so that the conversion of reactants into products is greater. In addition, according to Lili *et al.* [24] activated carbon has a high surface area and has resistance to various pH variations hence, the activated carbon catalyst can improve reaction performance well. In Figure 5, the relation between % total liquid yield function activated carbon (catalyst) concentration at 3 hours of pyrolysis time.

However, in the amount of catalyst of 2% in activated carbon catalyst, there was a decrease in the %total liquid yield due to the variable number of catalysts more and the higher temperature the more gas products were produced. The gas products are not only H_2 gas, but there are also other gases, such as: CO , CO_2 , CH_4 , C_2H_4 , and C_2H_6 [25].

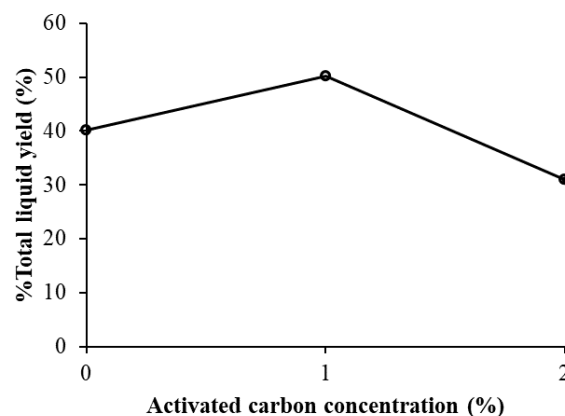


Figure 5. Relation graph between activated carbon (catalyst) concentration and %total liquid yield at 3 hours of pyrolysis time.

Table 3. Physical properties of biofuel (liquid product from 3 hours, 1% catalyst).

| Parameter | Test method | Heavy | Light | SNI 7182-2012 |
|---------------------------|-------------|-------|-------|---------------|
| Density (g/mL) | ASTM D 4052 | 0.88 | 0.65 | 0.85 – 0.89 |
| Kinematic viscosity (cSt) | ASTM D 445 | 5.79 | 2.32 | 2.30 – 6.00 |

3.4 Analysis of Biofuel

The physical characteristic of the *Chlorella sp.* biofuel in comparison with diesel fuel that conforms SNI 7182-2012. It is important to know in determining the quality of biofuel, especially density and kinematic viscosity. Based on the Table 3, the heavy fraction density is in accordance with the standard. However, the density of light fraction still does not fulfill the standard. This is because the light fraction of biofuels has alkane bonds that are too long and have few double bonds [26]. Meanwhile, related to kinematic viscosity is in accordance with applicable standards.

3.5 Fourier Transformed Infrared (FTIR) Analysis

3.5.1 FTIR analysis of heavy fraction liquid oil

The FTIR spectral of heavy fraction liquid oil (Table 4) showed the presence of amine, alkynes, alkenes and fluoro bonds at the peak vibrations. The test results show that the peak with high intensity is at a wavelength of 1632.42 cm^{-1} , namely the alkene group. In addition, the peak is at a wavelength of 3261.74 cm^{-1} , which can be interpreted as a moderate intensity range of vibrations of the N-H group, 1632.42 cm^{-1} , namely the alkene

group and 1408.75 cm^{-1} , namely the C-F range with low intensity.

3.5.2 FTIR analysis of light fraction liquid oil

FTIR analysis results of light fraction liquid oil from microalgae *Chlorella sp.* can be seen in Table 5. FTIR spectral shows the presence of carboxylic acid bonds, alkanes, isothiocyanates, cyclic alkenes, nitro compounds, methyls, alkanes, alkyl aryl ether, sulfoxides, halo compounds at their peak vibrations. The test results showed that the peak with high intensity was at a wavelength of 2955.82 cm^{-1} , namely the alkane group.

In this lighter fraction, some of the components show proportions with kerosene content (204–288 °C) due to the presence of alkanes (paraffins), sulfur compounds and cyclic hydrocarbons [28]. The presence of nitrogen compounds in this fraction is due to the nitrogen content in the microalgae *Chlorella sp.* of 12.13% so that it is possible to have a nitrogen content. Furthermore, the nitrogen content resulting from microalgae pyrolysis can include amines, amides, pyridines, pyrroles, pyrazoles, pyrazines, nitriles, imidazoles and indoles, although most of these compounds are amides [29]. The ester compounds are the result of the cracking of the

Table 4. FTIR spectral of heavy fraction liquid oil [26].

| Frequency Range (cm^{-1}) | Absorption (cm^{-1}) | Group | Compound Class |
|--------------------------------------|---------------------------------|--------------------------|-----------------------|
| 4000 – 3000 | 3261.74 | N-H | Aliphatic prime amine |
| 2400 – 2000 | 2161.18 | $\text{C}\equiv\text{C}$ | Alkyne |
| 1670 – 1600 | 1632.42 | $\text{C}=\text{C}$ | Alkenes |
| 1400 – 1000 | 1408.75 | C-F | Fluoro bonds |

Table 5. FTIR spectral of light fraction liquid oil [26].

| Frequency Range (cm^{-1}) | Absorption (cm^{-1}) | Group | Compound Class |
|--------------------------------------|---------------------------------|---------------------------------------|------------------|
| 4000 – 3000 | 3246.40 | $\text{O}=\text{C}-\text{O}-\text{H}$ | Carboxylic acid |
| 3000 – 2500 | 2955.82 | C-C | alkanes |
| 2140 – 1990 | 2010.53 | $\text{N}=\text{C}=\text{S}$ | Isothiosinate |
| 1650 – 1566 | 1654.39 | $\text{C}=\text{C}$ | Cyclic alkenes |
| 1600 – 1300 | 1514.18 | N-O | Nitro coumpund |
| 1600 – 1300 | 1455.39 | C-H | Methyl compound |
| 1275 – 1200 | 1268.20 | C-O | Alkyl aryl ether |
| 1210 – 1163 | 1166.68 | C-O | Esther |
| 1070 – 1030 | 1032.58 | S=O | Sulfoxide |
| 1000 – 650 | 740.86 | $\text{C}=\text{C}$ | Alkenes |

lipid content of 12.41% whereas carboxylic acid is deoxygenated cracking of lipid content [30]. In addition, the analysis of the elaboration of the following pathway postulates can be explained and verified in the characteristic analysis of the FT-IR (Figure 6).

4. Conclusions

Biofuel from *Chlorella sp.* can be produced by conventional pyrolysis process. The time and concentration of the catalyst have a significant effect on the produced biofuel. Longer pyrolysis time and higher catalyst concentration used would give the more product yields and optimum conditions obtained during the pyrolysis time of 3 hours and the use of activated carbon (catalyst) 1% with a total liquid yield of 50.20% (heavy fraction yield, 43.75% and light fraction yield, 6.44%). Moreover, the packed bed reactor with bubble column is the best choice for converting biofuel from microalgae because it

gives different fractions, so that it is easier to process further to the commercial biofuel stage.

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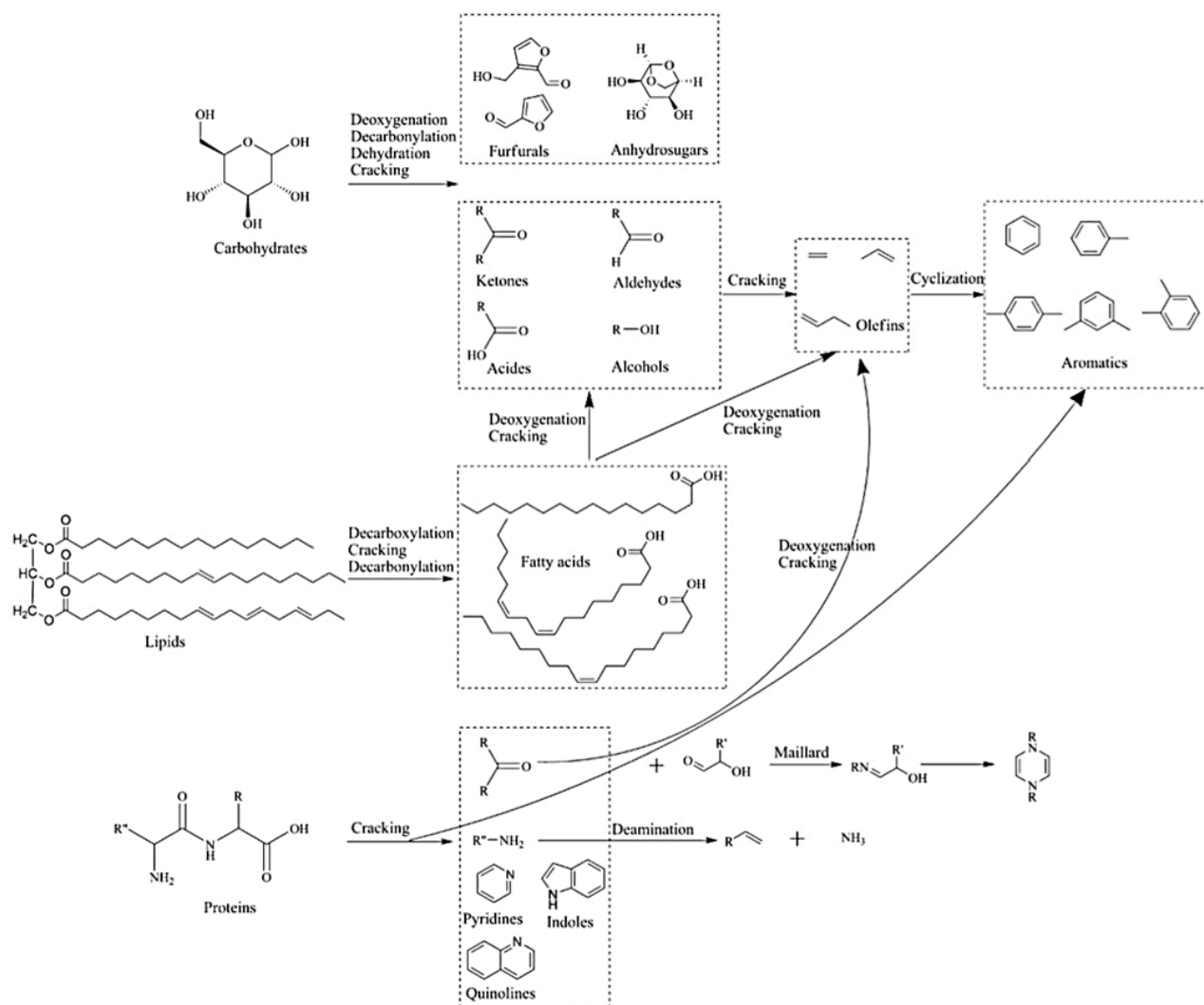


Figure 6. Postulat pathway pyrolysis from microalgae [29].

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